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INVESTIGATING TRIBOLOGICAL PROCESSES IN POLYMERIC COMPOSITES*

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ABSTRACT

The paper presents some results on investigation of the superficial layers of several polymeric composites intended for tribological applications. The evaluation of the tribological behavior is done with the help of SEM images, virtual images obtained by 3D profilometry and X-ray diffractometry.

KEYWORDS: tribology, polymeric composites, 3D profilometry, X-ray diffractometry, SEM, optical microscopy

1. The necessity of non-destructive investigations in tribology

One of the general trends is the development of the integrated systems, with high degree of automatization in industry including manufacturings, energetics and transports. These systems necessitate a closer control and are vulnerable to failures, reducing the failure consequences being nowadays a design criterion due to the associated risks for the economical organizations, environment, labour force and population and for avoiding financial and time deadlines losses. Failures and damages of the machine elements, especially those produced by tribological processes, have an undesirable high percentage in making the systems unavailable (Fig. 1).

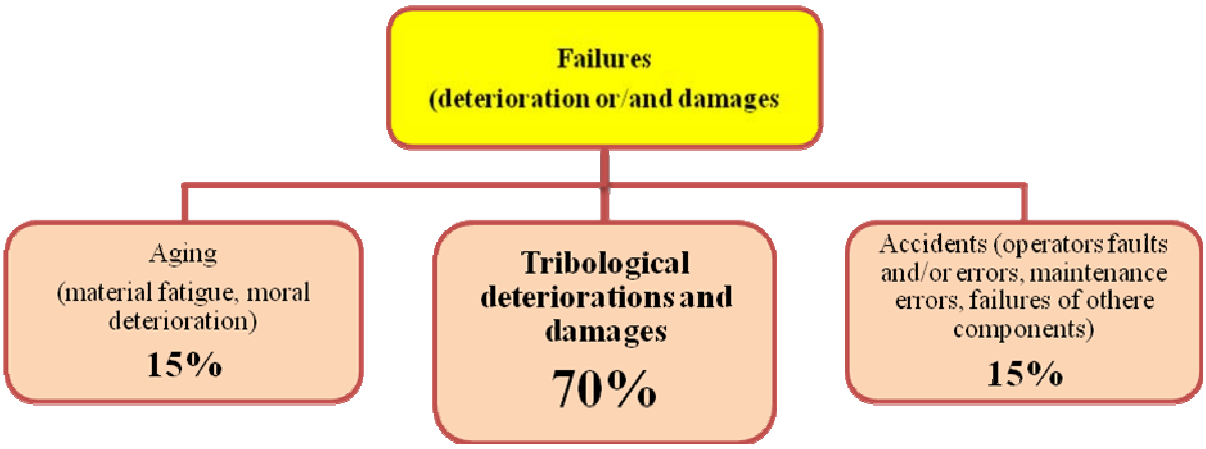

Fig. 1. *Percentage distribution of tribological deteriorations.*

Studies and researches in this field of tribology have a determinant influence in improving reliability and durability, starting from design but also in maintenance and diagnosis of the complex systems [1, 8, 41].

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There are generally admitted that the economic loss due to wear represents 6...10% from the gross national product of the developed countries and 30% of the failures of mechanical systems are caused by wear (Fig. 2) [1, 37].

The analysis of a tribological deterioration (Fig. 3) is usually included in a failure analysis or it could be the subject of an exploratory research in order to optimize the tribological behaviour of the system. Monitoring the tribological tests is difficult as it implies sophisticated and accurate equipment and alteration in a certain manner of the tribotester by introducing sensors [8, 9, 11, 15]. The tribologists have to prove inventivity for establishing a testing methodology with results that could be applied in practice.

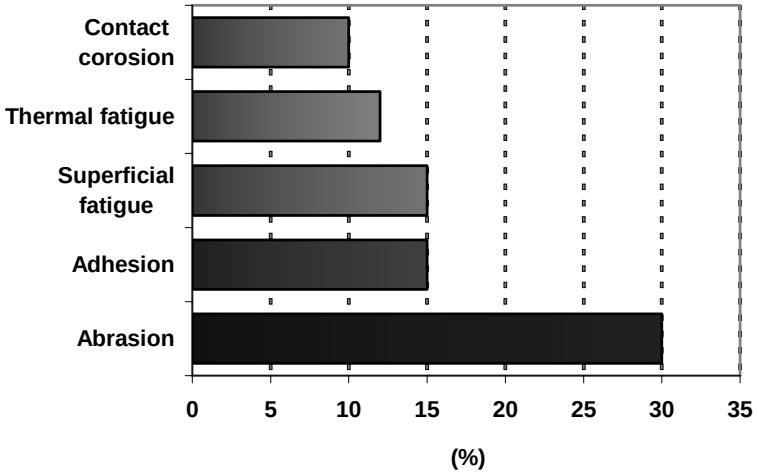


Fig. 2. *Percentage of different types of wear in the wear deteriorations.*

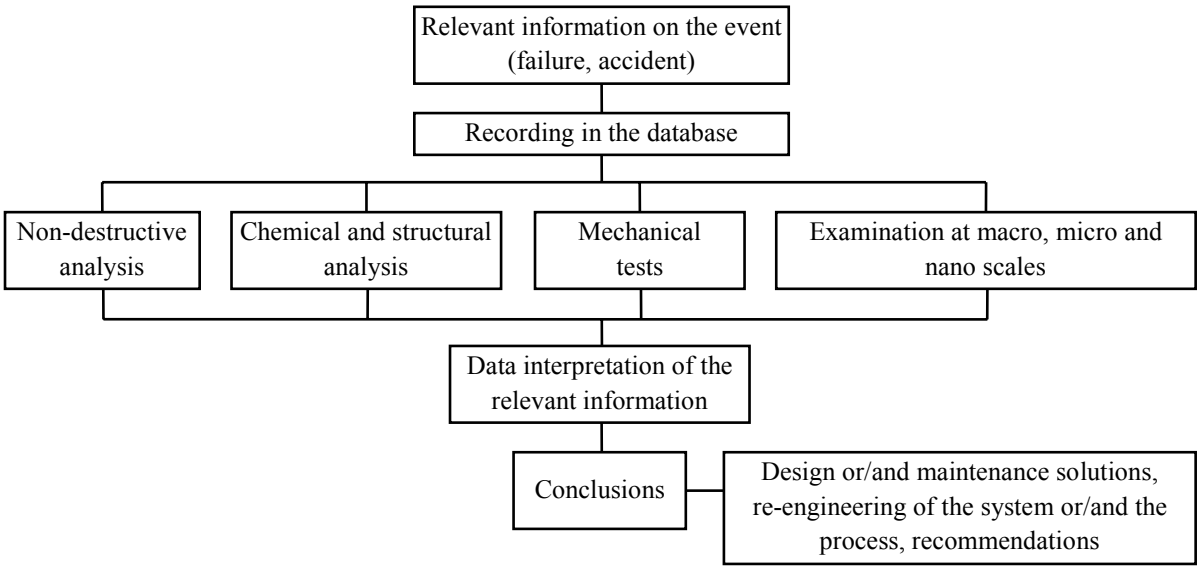


Fig. 3. *The chart for analysing a tribological deterioration.*

2. On polymeric composites

The polymeric composites are materials obtained by mixing different materials using a polymeric matrix: one or more adding materials could play the role of the reinforcement (Fig. 4) or/and of the solid lubricant. For other than mechanical applications the adding material could modify the physical, thermal and chemical response of the composite [2, 29].

Why are there so many grades of composites with polymeric matrix? The answer is given by the large ranges for the physical, thermal, mechanical, tribological properties of the constituent phases (two, three or even more) with very different concentrations. Even the polymer matrix could be

selected from many polymers [4, 7, 15] depending on the application. This diversity may offer solutions for a particular application taking into account the movement, the loading and speed regime, the lubrication, the working temperature, the environment.

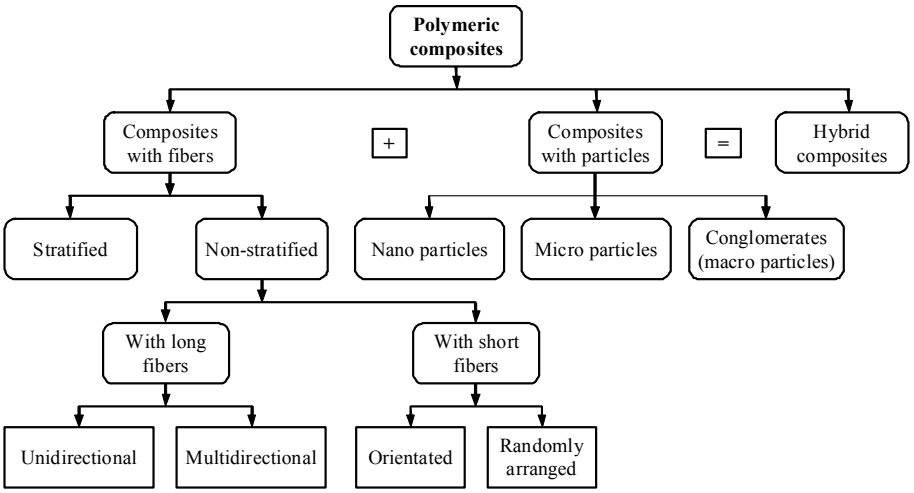


Fig. 4. *A classification of polymeric composites based on the shape and dimension of the adding materials.*

2.1. Tribological behaviour

The tribological behaviour of a polymeric composite is complex, including the tribological parameters and it has to explain the interconditionality of the influencing factors [5, 10, 29, 34, 37]. Tests on simpler triboelements such as pin-on-disk, block-on-ring etc., are easier to investigate and to identify the processes and the synergic influence of two or many factors.

The tribological behaviour means establishing relationships among influencing factors and tribological parameters such as friction coefficient, wear and wear rate, thermal field, structural and chemical changes, modifications of the physical and mechanical properties of the materials in contact, but also for the lubricant and the environment, changes of the surfaces topography etc.

2.2. Processes within the superficial layers of the polymeric composites

A synthesis of these processes is given in Figure 5 based on the recent references [6, 15-19, 25-26, 30, 34, 37, 38].

Taking into account the research field the authors are involved in [11-14, 20, 28, 39], the tribological processes of the polymeric composites are exemplified with the help of composites with PTFE and PA matrix, the polymers being kept as reference. PTFE [32, 33, 39, 40] and polyamide [8, 22, 27, 31, 35, 36] are two polymers frequently used as matrix due to their tribological behaviour.

3. Non-destructive analysis

Non-destructive analyses are preferable because they do not alter the structure or/and composition of the superficial layers [41], but there are limitations in monitoring the contact by non-invasive and non-destructive techniques.

3.1. Optical microscopy

This observation method has the advantage of easy preparation of the samples, sometimes they do not have to be cut from the tested pieces and nowadays there are available optical microscopes with high performances. One of the disadvantages is the magnification from 50...1400 (at least in our laboratory): a good resolution depends on the studied materials and on the quality of the worn surfaces. Sometimes scale like 200:1, 500:1 are preferable because they give images of the superficial layers allowing to understand the statistical characteristic of the processes. Figure 6 presents several PTFE composites and each image pointed out characteristics of the superficial layers. These scales are useful for evaluating, in a statistical and qualitative manner, the dominant processes within the superficial layers.

4. Scanning electron microscopy (SEM)

Scanning electron microscopy offers information about the sample's surface topography, composition and other properties such as electrical conductivity.

It has the advantage of high magnification 100...6000 (at least for the author's experience on polymers and polymeric composites and for the available equipment at our University).

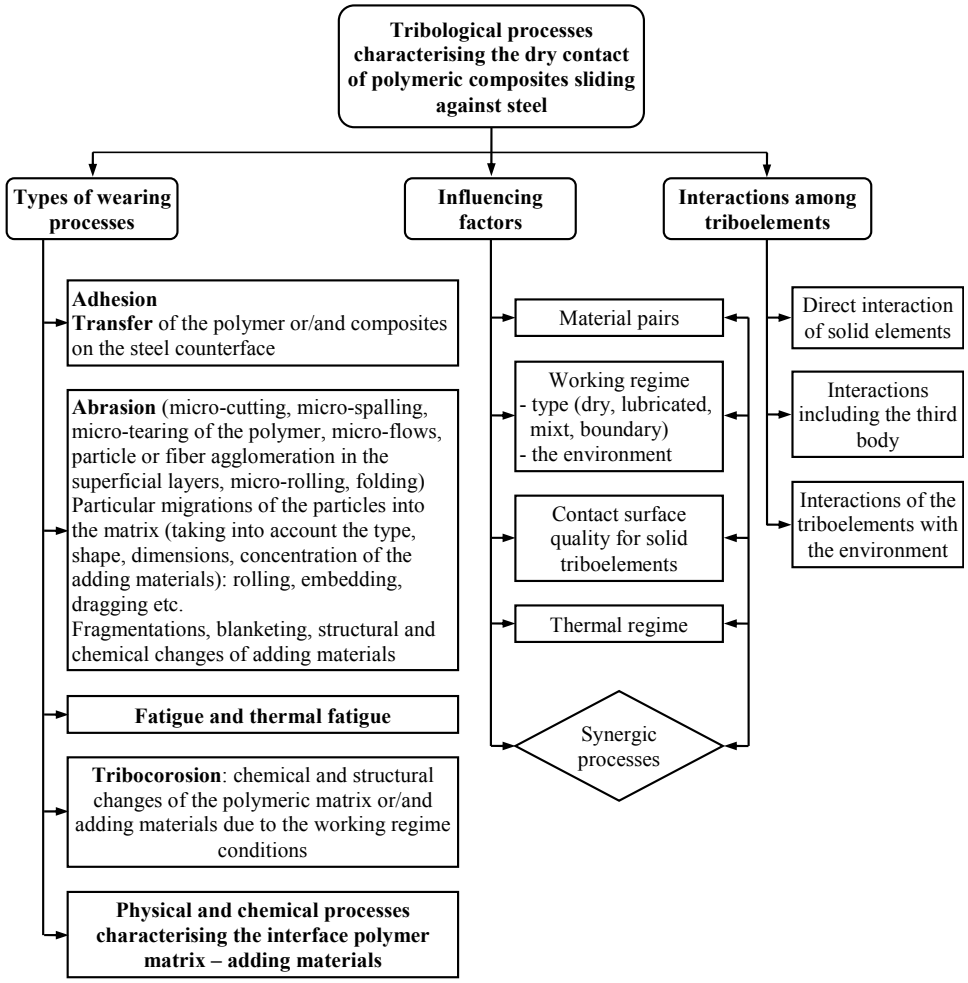
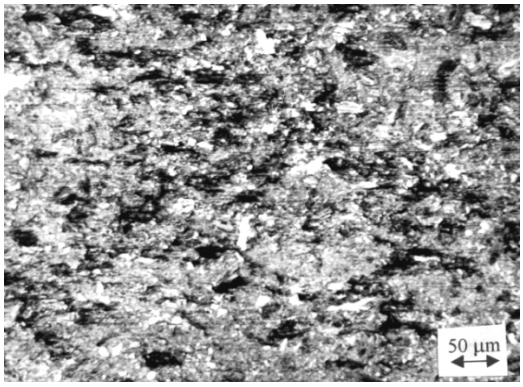
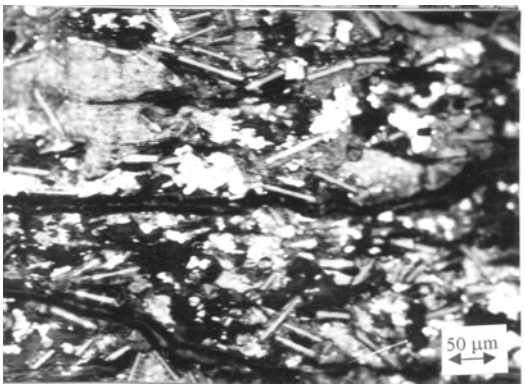


Fig. 5. *An overview of the tribological processes characterising the dry contact of polymeric composites sliding against steel.*

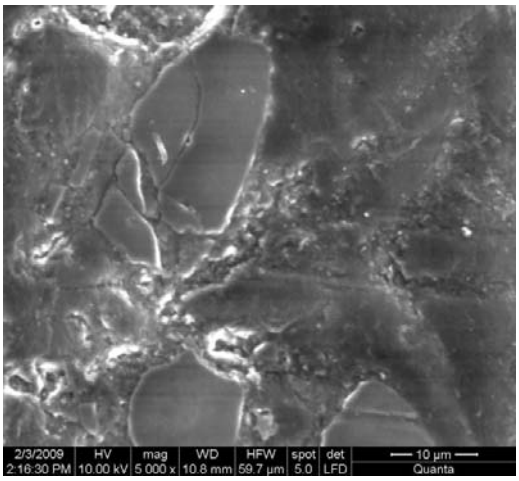


c) PTFE + 23% carbon + 2% graphite: micro flows of PTFE in the sliding direction and a mixing process of the polymer and carbon components into the superficial layer

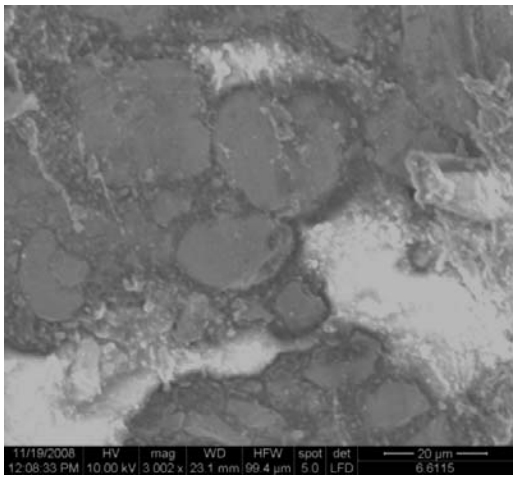


d) PTFE + glass fiber + graphite: fiber orientation in the sliding direction and micro flow of polymer and graphite mix

Fig. 6. *Dry sliding against steel, p =0.76 MPa, v =1 m/s Tribotester shoe/roller (Ra=0.8...1.2 µm) [13, 39].*

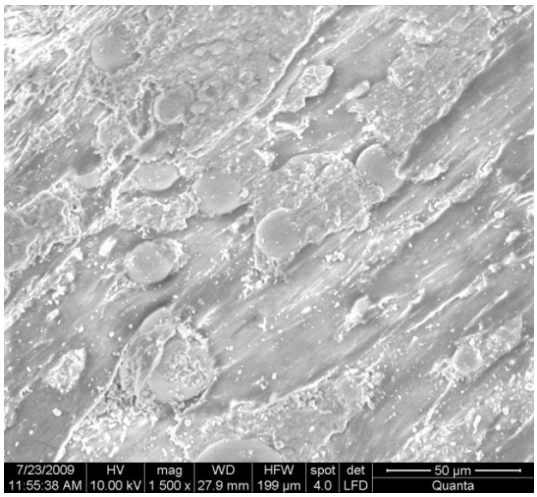


a) PTFE +15% glass fiber; particular processes: a broken glass fiber, fragments embedded in the PTFE matrix; local flow of the polymer and wear debris adhering among the fibers [13, 39]

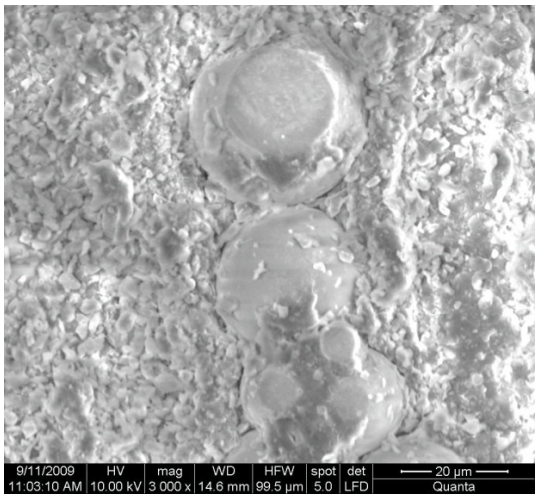


b) PTFE + 60% bronze; particular processes: very thin bands of polymer torn off from the matrix and spread on the hard constituent, explaining low friction and reduced wear [13, 39]

Fig. 7. *SEM images revealing processes influencing friction and wear of the tribosystem*



a) p=2 MPs, v=0.5 m/s



b) p=3 MPa, v=1.5 m/s

Fig. 8. *Influence of the sliding speed on the processes taking place into the superficial layers of the composite polyamide +20% micro glass spheres, after dry sliding of a steel pin on the composite disk [29].*

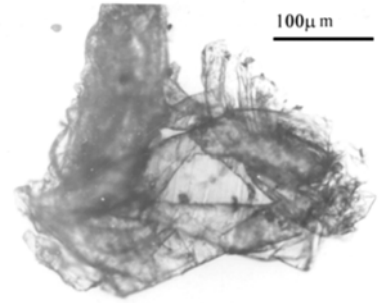


Fig. 9. *Wear particle from a shoe made of PTFE, dry sliding (p=0.76 MPa and v=0.5 m/s).*

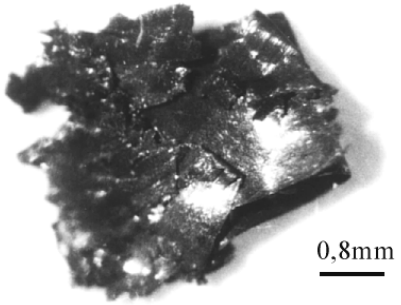
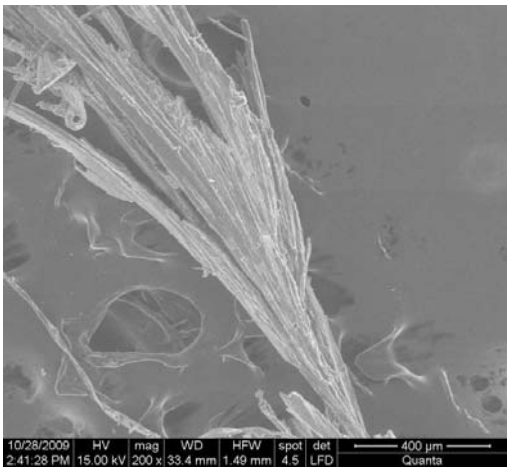
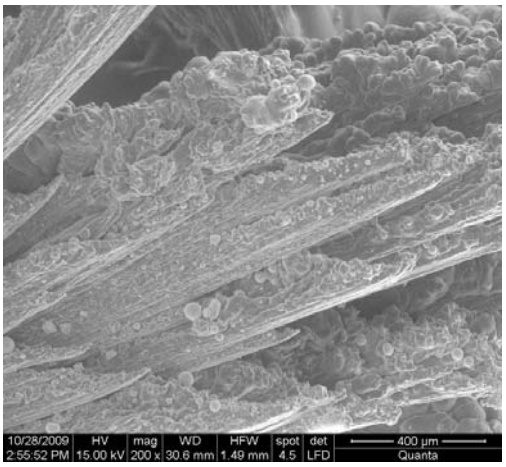


Fig. 10. *Wear particle from a shoe made of PTFE + 60% bronze, water lubrication (p=0.76 MPa and v=0.5 m/s).*



Wear particle obtained after dry sliding 10 km at v=1 m/s, p=1 MPa, pin-on-disk, disk made of polyamide, pin made of steel (background - carbon strip)



Wear particle obtained after dry sliding 10 km at v=1 m/s, p=1 MPa, pin-on-disk, disk made of composite with polyamide matrix and 20% micro glass spheres, pin made of steel

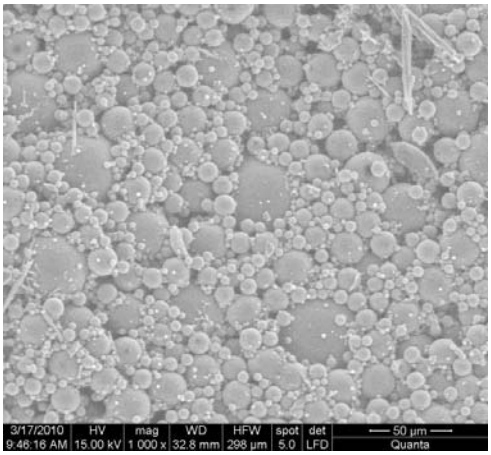
Fig. 11. *SEM images of debris detached after pin-on-disk tests.*

The disadvantages are that it is a time-consuming investigation and special higher resolution coating techniques are required for high magnification imaging of either organic or inorganic thin films.

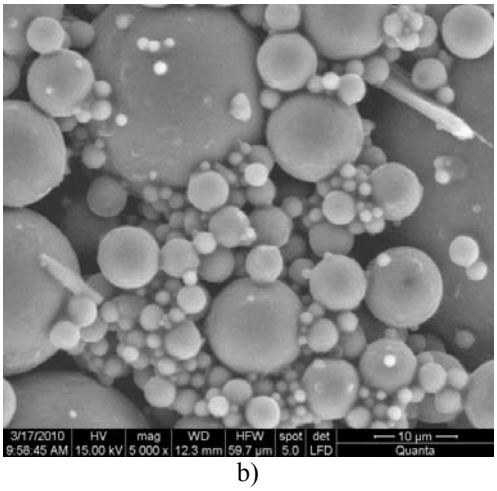
The images could reveal how a glass fiber is broken (Fig. 7a) or how the soft polymer such as PTFE is acting like a solid lubricant (Fig. 7b).

The symbol *p* is used for average pressure on contact.

Both optical and SEM microscopies allow studying the wear debris and the influence that some factors such as regime type, speed and load could influence their generation. SEM and optical images could be useful for estimating the size distribution of the adding materials, but the results are sensitive to the scale and the number of the analyzed images. For instance, Figure 12 presents two SEM images of micro glass spheres that will be added into a polyamide matrix. Using an appropriate soft, the images at a smaller scale could give a more accurate size distribution but a larger scale reveals the smaller particles and their size around several hundred nanometers.



a)

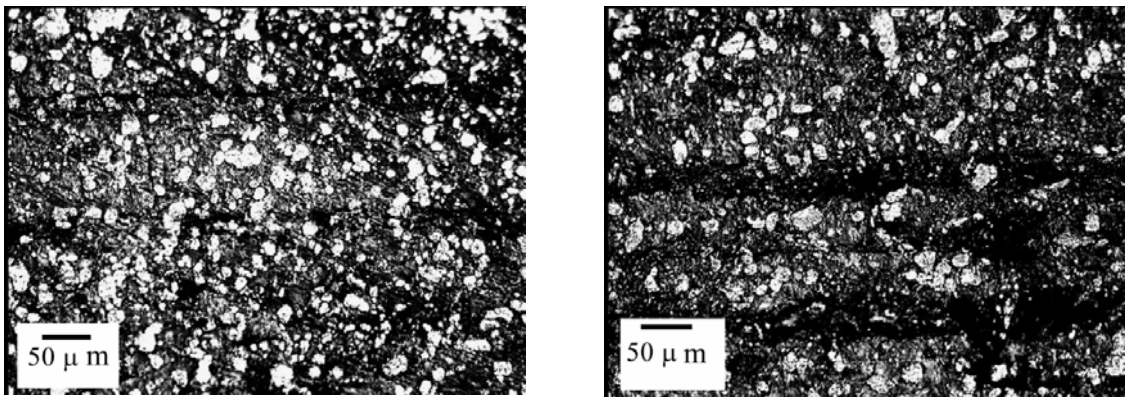


b)

Fig. 12. *SEM images of the micro glass spheres, at different scale that could be used for evaluating the size dispersion.*

If the composite constituents are chemically non-reactive one to each other during the functioning of the tribosystem, the images could serve to evaluate the surface concentration of each one. For instance, in dry regime for the composite PTFE + 60% bronze, it was obtained a surface concentration of 15.7...21.5% PTFE. The bags of PTFE have the tendency to get longer in the sliding direction and the process of

polymer transfer is observed even on the composite (Fig. 7b) not only on the steel roller, in thin slices (light-grey colours); this process is favourable for reducing friction as presented in the model [5, 37]. After sliding in water, the surface percentage of PTFE on the tribolayer is 11.35...18.35% for the studied shoes, similar to the other shoes tested in water in open circuit at speed higher than 1m/s. For this composite, PTFE is captive and isolated in the metallic mass and this is the reason why the surface concentration on the tribolayer is not significantly changed. PTFE bags have the tendency to become spheres under the action of stress field, maybe with flat or cut zone on the surface. These spheres are smaller towards the surface as the tribolayer is locally compressed and fragmented (Fig. 13). In dry regime it was obtained a surface concentration of 15.7...21.5% PTFE.



a) the highest concentration obtained for the shoe tested at 4 kN

b) the least concentration on the same shoe, obtained in the middle zone of the loaded zone

Fig. 13. *Surface of the composite PTFE + 60% bronze after sliding in water for 10,500 m at v=2.5 m/s and F=4 kN, test done on shoe ∅60 x 25 mm on steel shaft.*

3.3. X-ray diffractometry

X-ray diffraction techniques could do a characterization of crystalline materials, including polymers, and their composites, identifying the phases present in samples from raw materials to finished products and to provide information on the physical state of the sample, such as grain size, texture, and crystal perfection. Most X-ray diffraction techniques are rapid and nondestructive [20, 23]. By X-rays diffraction method, the structure and inner tension changes occurring in the superficial layer have been evinced.

Wide angle X-ray diffractometry provides information on the crystal structure and the orientation. Low angle X-ray scattering offers information on the size and regularity of lamellar crystals' packing but the interpretation is often a difficult matter [23]. A systematic study on the tribological behaviour of polymers and their composites is hard to achieve during testing; thus many results are dealing with the analysis of after-tests.

After dry friction, the X-ray investigation of the structural changes in the superficial layer of PTFE, the volume of the hexagonal cell was found to be reduced, the crystallites were found to be enlarged and residual stresses were found to be introduced as a result of this transition [21, 40]. Figure 14 present diffractograms in a range of 30…45 degrees for the angle 2θ as the most important structural changes of this polymer occur in this angle range [20, 41]. Diffracted X-rays are mono-chromatised before being detected, this leading to avoid background noise.

After friction under lubricated regime (water), the amorphous structure is more consistent. The fragmentation of the molecular chains of PTFE is pointed out by the appearance of new diffraction peaks. This is caused by mechanical processes as a consequence that water film is discontinuous.

The same cause also provokes a forced orientation of the fragmented micro-volumes and chains of the polymer within the superficial layer, pointed out by the increase of peaks' intensities. For v=1.5 m/s, within the superficial layer, the fragmentation of the molecular chains is diminished as compared to that for v=0.7m/s, but the noticed fragments have a more accentuated orientation. This could be explained by the fact that there are conditions to generate a more stable lubricant film and on a greater part of the contact area.

For the composites PTFE + glass fibers, the same types of structural modifications are noticed (fragmentation and forced orientation of the molecular chains of the polymer) but they are less intense, and the modifications diminish as the glass fiber concentration increases.

As pointed out in [5, 33, 37, 39], the glass fibers play the part of an unorganized net that does not allow

for a too intense fragmentation and orientation of the polymer matrix within the superficial layers, also reducing the wear with one order or even more [6, 19, 39].

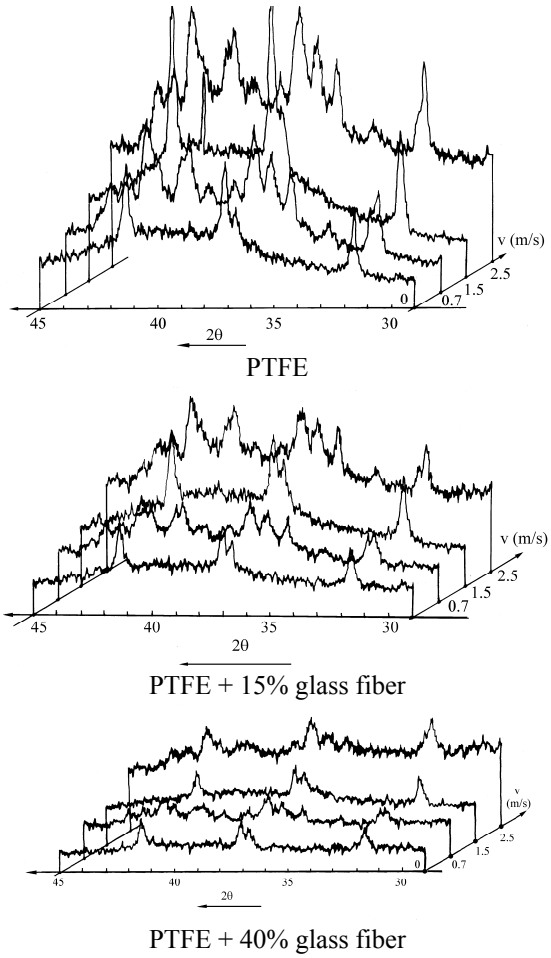


Fig. 14. *The parameters of the investigation were voltage U=34kV, current intensity I=30mA and there was investigated a range of 2θ between 25 and 45 degrees.*

After testing by sliding in water at constant speed and load, against steel, the new appeared structures in composites PTFE+ glass fiber are in a lower quantity as comparing to the pure PTFE sample. This change in the diffractograms may be explained as following:

- the presence of a smaller quantity of PTFE in the superficial layer due to adhesive or abrasive wear, supported especially by the polymeric matrix,
- the presence of glass fibers does not probably allow orientating the new structures.

Comparing after-testing diffractograms for PTFE +15% glass fiber to that for PTFE + 40% glass fiber, one may notice the presence of the same structures but lower fiber concentration. The PTFE peaks are very clear and the structure of the polymer is much more oriented as compared to the composites.

An increase of the peaks' heights for higher speeds may suggest a forced orientation of the polymer crystallites.

But for the highest value of sliding speed, the peak heights decrease and the basic level of the diffractograms is lifted, pointing out a decrease of the structure orientation and an increase of the amorphous phase. These aspects may be interpreted only making a correlation to wear values. The depth of the X-ray investigation is approximately 100…200 μm. If wear is considerable, the superficial layer investigated for extreme values of sliding speed is totally new, and very fragmented. It is important to point out that average pressure is not quite relevant for result interpretation.

It will be more useful to know the maximum contact pressure that could be several times greater than the average, closer to the flow limit of the polymer. For composites PTFE + glass fibers, the changes in diffractograms aspects are not so dramatic, suggesting that the fiber net makes the superficial layer more stable, including the structure orientation and the crystallinity degree. The higher level of the after-test diffractograms indicates the fragmentation of initial structures.

The appearance of new structural crystallites is not so evident for composites with higher fiber concentration.

Pointing out these aspects allows selecting the adding material related to their quality (fibers, powder, mechanical properties etc.) and their quantity in order to obtain a composite with a better tribological behaviour.

3.4. 3D profilometry

Due to modern softwares, the data recorded with the help of 3D contact or non-contact profilometers, make possible to calculate many roughness parameters [3, 42].

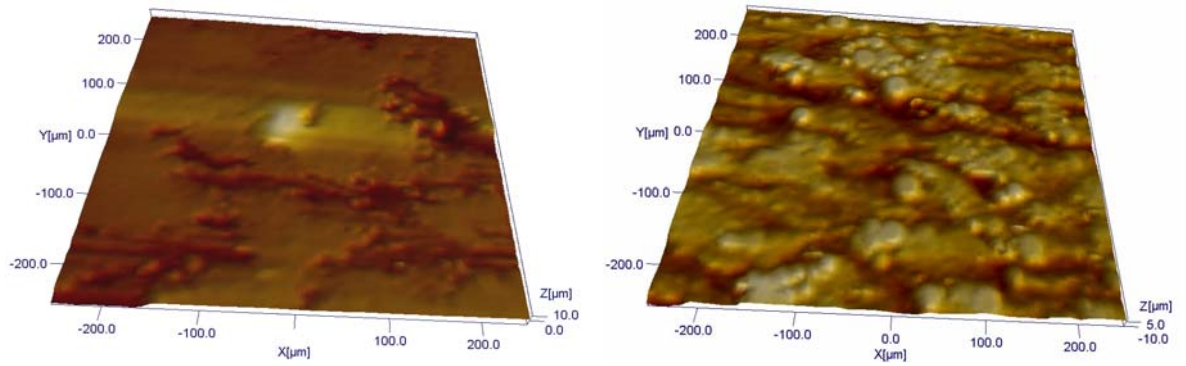
Comparing statistical data on these parameters, the tribologists could establish the surface quality after testing and the influence of several factors on the magnitude of parameters.

They are especially interested in studying the functional parameters (as given from the Abbot Firestone loading bearing area).

Virtual images as those in Figure 15 could be used for identifying the processes taking place into the superficial layers.

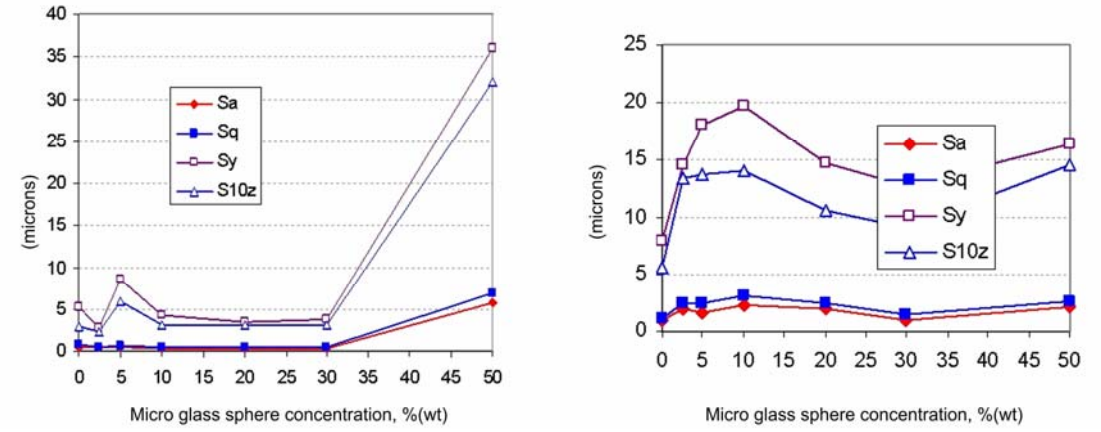
Figure 16 presents the influence of the micro glass spheres concentration on the amplitude parameters for two dry sliding regimes. The interpretation should be done carefully as the plots have a statistical character, and could be altered by the

number of measurements [12, 28]. In this figure, the plotted results are the average value from three measurements on 500 x 500 μm, equi-angularly on the wear track of the composite disk.



a) v=1 m/s, p=1 MPa *b) v=1.5 m/s, p=1 MPa*

Fig. 15. *Virtual images created from data obtained with the help of a dedicated soft [42] after data acquisition with the help of a 3D profilometer for the wear track on the composite with polyamide matrix and 50% micro glass spheres, after being tested under dry sliding (pin-on disk tribotester).*



v = 0.5 m/s *v = 1.5 m/s*

Fig. 16. *Amplitude parameters as a function of concentration of micro glass spheres for two sliding speeds, at p=3 MPa (all symbols are defined in [3, 42]: Sa – roughness average, Sq – root mean square, Sy – the maximum height peak-valley, S10z – ten point heights.*

3.5. Thermal survey

This method of monitoring the contact during functioning is now easy to be done with the help of an accurate thermographic camera and a dedicated soft for obtaining the temperature evolution in time on different zones of the triboelements „exposed" to the camera.

Figure 17 presents a thermographic survey of a pin-on-disk contact for a composite PA+ micro glass sphere revealing that thermal field and the friction coefficient are interconnected.

Using analytical or numerical methods and knowing the temperature at the contact edge, one may calculate the maximum temperature in contact.

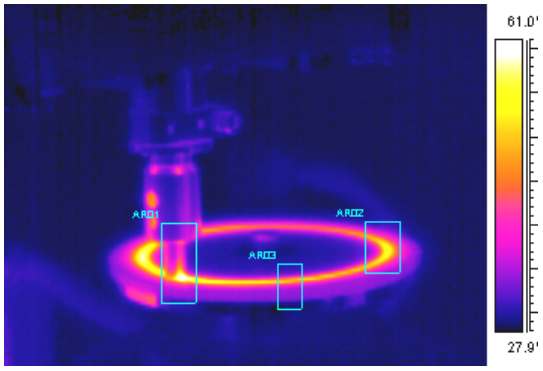


Fig. 17. *Points for temperature survey for a pin-on-disk test for a disk made of polyamide and pin made of steel.*

The temperature is very important for the polymeric composite [2, 5, 37], allowing a decrease of the friction coefficient when the temperature is high enough to generate a very thin viscous or melt polymer film. The monitoring of the temperature field was done with the help of a thermographic camera. Figure 18 gives the temperature evolution for three points on the pin-on-disk tribotester: AR01 – for the contact between pin and disk, AR02 – 90 degrees in back of the contact, on the wear track, AR03 – a point diameterlly opposed to the contact. Studying the friction coefficient one may notice that peaks of temperature evolution are accompanied by rises in the friction coefficient, meaning a change of the regime conditions.

A possible explanation is: a high temperature softens the polymer and for a time period, this viscous material help reducing the friction coefficient value and the process is dynamically repeated, with different time durations.

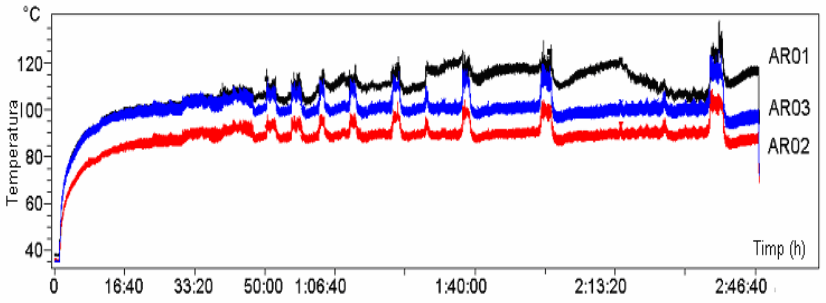


Fig. 18. *Temperature recorded in three points on the wear track of the disk made of composite with polyamide matrix and 20% micro glass spheres at v=1 m/s, p=1 MPa [28].*

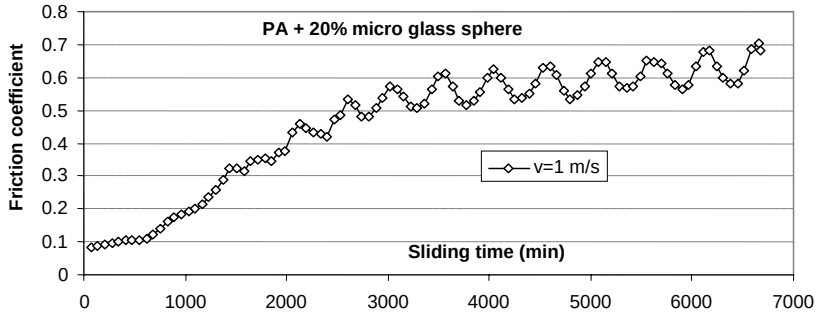


Fig. 19. *Evolution in time of the friction coefficient for the same test conditions as those in Figure 18 [28].*

4. Conclusion

The investigation of the superficial layers of the polymeric composites has to use several methods (some of them being exemplified in this paper) in order to identify the processes and the influencing factors.

Of course there are other investigation techniques that were not mentioned there (AFM microscopy, spectrometry etc.).

Specialists have to design the adequate set of investigation techniques, that could reveal the relevant processes for tribological applications.

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